

Single and combined effects of process parameters on biodiesel produced from blends of palm kernel shell and cocoa pods oils with titanium oxide as catalyst

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Global Journal of Engineering and Technology Advances, 2026, 28(01), 096–111

Publication history: Received on 04 June 2026; revised on 12 July 2026; accepted on 15 July 2026

Article DOI: <https://doi.org/10.30574/gjeta.2026.28.1.0177>

Abstract

Effects of process parameters on biodiesel produced from blend of palm kernel shell (PKS) and cocoa pods (CPO) oils, is investigated. These bio-oils were transesterified with methanol in the presence of titanium oxide as catalyst. The study shows a significant effect of the process parameters on the properties of biodiesel and its yield. The process parameters - catalyst concentration, reaction time, reaction temperature, methanol/sample ratio and agitation speed - were studied individually and in matrix form to establish synergistic interactions. Catalyst concentration of 5wt%, methanol/sample ratio of 6:1, agitation speed of 250rpm, reaction time of 90 minutes and temperature at 60°C was the optimal process conditions that gave the maximum yield of 76.05%. Other set of conditions that resulted the lowest biodiesel yield of 34.22% were catalyst concentration of 1.5%, methanol/sample ratio of 4:1, agitation speed of 150rpm, reaction time of 30 minutes and temperature of 40°C. The physiochemical properties of the yield investigated to ascertain their adherence with the American Society for Testing and Materials (ASTM) and European Nation (EN) standards for biodiesel production revealed that the developed biodiesel properties conformed with the recommended standards. Response Surface Methodology (RSM), which identified the various numerical and discrete design points was employed to achieve the optimum process parameters for the production of biodiesel using titanium oxide as catalyst. ANOVA statistics was also used to predict the biodiesel yield and to understand the combined effects of the process parameters. For single effect, result shows that amongst the five (5) variables studied, the methanol/oil (B) had the largest effect on the biodiesel yield as it took the highest F-value (11.26) while the least was the reaction time (D) with the lowest F-value of 0.2225. For the combined effect, methanol and temperature (BC) had the highest effect on the biodiesel yield as it took the highest F-value (12.01) while the least was catalyst concentration and time (AD) with the lowest F-value (0.0016).

Keywords: Bio-oil; Process parameters; Single effect; Combined effect; Biodiesel yield; Titanium oxide catalyst

1. Introduction

Fossil fuels play a very important role in industrial growth development, transportation sector, agricultural sector, and other global energy demand sectors [1]. Its importance cannot be underestimated. It constitutes the single largest energy source in the entire world; which accounts for about 88% of the global energy consumption [2, 3]. The continuous usage of crude oil products for energy generation has caused the depletion of oil reserves and this has led to the increase in the prices of petroleum products. The increased usage of fossil fuels for transportation and energy has also resulted in global warming and ozone layer destruction and health risks for people worldwide [4, 5, 6]. This is as a result of the increased emission of toxic gases like CO₂, SO₂, etc, into the atmosphere due to the combustion of fossil fuel [3, 7, 8]. As the fossil fuels are depleting day by day, and the global energy crisis is of concern, there is a need to find out an alternative fuel to fulfill the energy demand of the world [1, 9]. This alternative energy sources must be

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renewable, eco-friendly, available, efficient, and viable, sustainable and economical [3, 7, 10, 11]. For this reason, alternate energy sources are currently being researched in order to reduce environmental carbon footprints and to increase energy efficiency [12, 13]. Biodiesel has proven to be one of the alternative fuels that can substitute fossil fuels [1, 3, 7, 12]. They could be generated through variety of processes like, physiochemical (eg. transesterification, and esterification), thermochemical (eg. pyrolysis, and gasification), and biochemical (eg. anaerobic digestion, fermentation etc.) [7, 14]. However, trans-esterification has been viewed as an excellent process for biodiesel production, because it helps in removing the glycerides present in the bio-oil, thereby allowing the oil esters of the vegetable oil to mix with alcohol [7, 15]. It also helps reduce the high viscosity of the bio-oil [3]. Biodiesel produced from oily feedstocks necessitates alcohol and catalyst in the transesterification process [16, 17]. The catalyst (homogeneous or heterogeneous) is used in biodiesel production in order to accelerate the reaction time, and increase oil yield. This is done in the presence of alcohol (methanol or ethanol). In this work, heterogeneous catalyst (titanium oxide) was chosen, because of its low cost, reusability, less corrosive nature, eco-friendliness, ease of separation etc., over the homogeneous catalyst [7, 16, 17, 18]. Methanol was also preferred because of its availability, low cost etc. Researchers have carried out various works in the quest to address the issue of poor quality biofuels, low yield, high cost, ease of production etc. These have resulted in the use of various feedstocks, catalysts, and methods of production [3, 7, 16, 18, 19]. However, studies on the use of palm kernel shell oil or cocoa pods oil or its blend with titanium oxide as catalyst have rarely been considered or recorded; despite their abundance in the sub-Saharan Africa. This study focuses on the use of blend of PKSO and CPO as hybrid feedstock in the production of biodiesel with a nano catalyst (titanium oxide) through transesterification process; and the analysis of single and combined effects of process parameters (reaction time, methanol/oil ratio, agitation speed, temperature, and catalyst concentration) on percentage oil yield. There are different software tools that could be used for predicting and optimizing biodiesel yield [9, 18, 20, 21, 22, 23]. In this study, Response Surface Methodology (RSM) was used to predict, and model the transesterification process owing to its simplicity, and ability to correlate the input variables with responses; thereby achieving increased oil yield, and reduced production cost. This fact was already recorded by [3, 7, 16, 23].

2. Literature review

When considering the production of biodiesel, the primary substances are the feedstocks. These feedstocks are classified into first, second, third and fourth generation feedstock. Oils extracted from these sources for the production of biodiesel fall under edible and non-edible oil. They could be used as single feedstock or hybrid (combined). Biodiesel made from food-grade vegetable oils (edible oils) is too expensive to compete on cost basis with fossil-based diesel, as it would reduce the global food bank [16, 18]. As a result, the non-edible agro-source oils are preferred as feedstocks for biodiesel production (BPR) because they are regarded as agricultural waste product [13, 16]. Nigeria and other Sub-Saharan African countries have abundance of the major agro wastes used as feedstocks for biodiesel production [3]. Examples are palm kernel shell and cocoa pods used in this work. They are underutilized, and have become nuisance to the community [24]. Wastes generated by the global palm oil industries, for example, contain more than 70% PKS, making disposal difficult [25]. The same is applicable to the cocoa pods. Akinola et al. 2018 [26] reported that oil can be extracted from cocoa pods and used for the production of biodiesel. This gives solution to the most effective use of PKS and CP, thus keeping them from becoming nuisance to environment. According to [16 and 25], the palm kernel shell oil's suitability for biodiesel production is mostly attributed to its superior fuel properties and prominent lauric feature compared to average lignocellulosic biomass. Other works [7, 16, 25, 27], have testified to high quality biodiesel and better yield from palm kernel shell oil. Work by Lee and Ofori-Boateng, (2013) [28, 29] also used cocoa pods as renewable source for developing catalyst for biodiesel production; but reports on the use of the two as hybrid for biodiesel production (BPR), with titanium oxide as catalyst have been limited.

Basically, different researchers have carried out various research using different feedstocks, catalysts, and processes for BPR. With respect to the above factors and process parameters considered, the percentage oil yields differ. Table 1 presents the summary of selected literature of works done by some researchers and their results.

Table 1 Summary of selected related literature and results obtained

Reference	Feedstock	Reaction Time (min)	Biodiesel Production Process	Bio-processing Temperature (°C)	Molar to Oil Ratio	Type of Catalyst	%Yield
Agarry <i>et al.</i> (2013)	Tangerine, orange and grape		Transesterification			potassium hydroxide	90.6% from the grape oil compared to the other citrus peel oils
Onukwili <i>et al.</i> (2016)	refined cotton seed oil	60	Transesterification with methanol	55	6:1	potassium hydroxide (KOH)	96%
Widayat and Suherman (2012)	rubber seed oil		Esterification				50.5%
Mishra <i>et al.</i> (2015)	crude neem oil	75		50	4:5:1	KOH	72 ± 2%
Aworanti, Ajani and Agarry 2019	Waste Frying Vegetable Oil (WFVO) and Waste Frying Palm Oil (WFPO)	90	Transesterification	60	12:1	KOH	97% and 90%
Ojolo, Adelaja and Sobamowo 2012	Palm kernel oil and groundnut oil	120	Trans-esterification	55		Methanol and sodium hydroxide pellets	91.98% for groundnut oil 90.53% for Palm kernel oil.
Sajjad <i>et al.</i> , 2022	Tamarindus indica Seed Oil	90	Transesterification	50	9:1	NaOH	93.5%

3. Methodology

3.1. Material collection, preparation methods and oil extraction

The major materials utilized for this study were carefully selected locally, to enhance reduction in biodiesel production cost. This was intentionally done due to the high cost of food resources, which is as a result of the competition between the use of edible oils and other food produce as biodiesel feedstock, as recorded by [36]. The study considered the extraction of oils from readily available agro waste produce (palm kernel shell and cocoa pods) for the production of biodiesel. Palm kernel shell and cocoa pods were sourced from local farmers in Akwa Ibom and Cross River States of Nigeria, respectively. The impurities were carefully selected out allowed to dry under the sun according to [37, 38, 39]. After, they were ground to powder form. Oil extraction was carried out, using a 500ml Soxhlet extractor; and characterized for density, refractive index, moisture content, saponification value, viscosity, to ascertain its suitability for the production of biodiesel. Other materials used include, beaker, heater, thermometer, retort stand, round bottom

flask, reflux condenser, separating funnel, weighing balance, litmus paper, nano catalyst (titanium oxide), distilled water, magnetic capsule, n-hexane, carbonizer etc.

The nano catalyst, titanium oxide, was purchased from Juiles Therapeutic factory in Lagos, Nigeria. After screening in a 500 μm particle size screen, it was submerged in distilled water for 120 minutes. The lower section was separated from the unwanted particles, which float on the water; and the catalyst was placed in an oven set to 600C for 120 minutes in order for it to dry.

After the oil extraction via soxhlet method using the flat bottom round flask with n-hexane, the raw sample in the semi-permeable membrane was disposed; and the n-hexane mixture and oil remaining in the flat bottom flask were separated by distillation process. During this process, the n-hexane was distilled over while the oil remained in the flask. The oil was characterized for density, refractive index, moisture content, viscosity, acid value, peroxide value, iodine value etc; and the results obtained was within the ASTM and EN recommended standard.

3.2. Biodiesel Production

3.2.1. Transesterification

Production of the methyl ester (biodiesel) was carried out in the 500 ml three necked round bottom spherical reactor through transesterification process. The quantity of methanol and catalyst (titanium oxide) needed for each round of the experiment were calculated. They were mixed together and stirred for 5 minutes to ensure proper dissolution. After that, 50 ml of the extracted blend (PKS and CP oils) was added to the mixture. A reflux condenser with an outer jacket for the circulation of water was fitted to the three necked reactor at the middle neck. A thermometer (mercury in glass) was fitted to the right neck. The thermometer was held to the reactor with a plastic bung. After that, a stirring nob was put into the reactor through the left neck; and closed with another plastic bung.

The setup was placed on a magnetic heating mantle and switched on; and the stirring system thereafter was switched on and maintained at an established speed and regulated at the required temperature. Each reaction was allowed to last for the required 'time' at specific 'temperature'. After the transesterification reaction, the product was allowed to stand for twelve hours in a separating funnel for glycerol separation. The crude glycerol was removed through the funnel tap leaving the methyl ester (biodiesel) behind.

3.3. Effect of process parameters on the produced biodiesel yield

The effect of process parameters (independent variables) which included catalyst concentration {wt. %}, reaction time (min), reaction temperature {degree Celcius}, methanol/oil molal ratio and agitation speed {rpm}, were studied individually (single effect) and in matrix form (combined effect) to establish synergistic interactions. The process parameters were varied at different points within the specified study range while all other parameters were kept constant.

3.3.1. Effect of catalyst concentration

The effect of the catalyst concentration on the yield of biodiesel was studied. Catalyst concentration in weight percent was varied at points: 0.5, 1, 1.5, 2 and 2.5 weight percent. At this study level, other parameters were held constant at temperature of 600C, methanol / sample mole ratio of 12:1, reaction time of 5 hours and agitation speed of 350 revolutions per minute (rpm).

3.3.2. Effect of reaction time

The reaction time was varied at 1, 2, 3, 4, and 5 hours; while other conditions were held constant at: catalyst concentration with maximum yield in i, temperature of 600C, methanol/sample mole ratio of 12:1 and agitation speed of 350 revolutions per minute (rpm).

3.3.3. Effect of temperature

Temperature was studied at different heating points: 40, 45, 50, 55 and 600C. Other conditions were held constant at: catalyst concentration with maximum yield in i, reaction time with highest yield in ii, methanol/sample molal ratio of 12:1 and agitation speed of 350 revolutions per minute (rpm).

3.3.4. Effect of methanol/sample molal ratio

The effect of methanol/sample molal ratio was studied at various concentrations of 4:1, 6:1, 8:1, 10:1 and 12:1. Other conditions were held constant at; catalyst concentration with maximum yield in i, reaction time with highest yield in ii, temperature with maximum yield in iii, and agitation speed of 350 revolutions per minute (rpm).

3.3.5. Effect of agitation speed

The speed at which the sample was stirred (rotation speed) in the reactor was varied at various points ranging from: 150, 200, 250, 300 and 350 revolutions per minute (rpm). All other determinant variables studied above were held constant at maximum points at: catalyst concentration with maximum yield in i, reaction time with highest yield in ii, temperature with maximum yield in iii, and methanol/sample molal ratio with highest yield in iv, respectively.

4. Results and discussion

4.1. Characterization of bio-oil

The blended bio-oils gotten from cocoa pods and palm kernel shell were characterized to predict the actual condition of the feedstock; if it will produce quality biodiesel. The results of the proximate and ultimate analysis obtained are presented in Table 2.

Table 2 Proximate and ultimate analysis of the blended feedstock oils

Parameters	Blended feedstock oil value
Refractive index @ 29°C	1.520
Energy value (kJ/kg)	140.5
Fire point (°C)	276
Cloud point (°C)	-14
Acid value (mgKOH/kg)	1.24
Saponification (mgKOH/kg)	194
Peroxide value (mEq/kg)	3.7
Molecular weight (g/mol)	701.45
Volatile matter	64.3

From the table 2, the refractive index @ 29 °C, energy value, fire point, cloud point, acid value, saponification value, peroxide value, molecular weight etc., are 1.520, 140.5, 276, -14, 1.24, 194, 3.7, 701.45 respectively. These results are in line with the standard bio-oil specification range for biofuel production.

4.2. Characterization of catalyst

The nano catalyst, titanium oxide, was also characterized, and the results obtained shows that titanium oxide is a heterogeneous catalyst with high potentials due to its high surface area, strong metal support interaction, chemical stability, and acid–base property. It proved to be very effective in the production of biodiesel.

4.3. Effects of process parameters on the biodiesel yield using nano catalysts

4.3.1. Single effect

The results of the single effects of the process parameters on the biodiesel yield using the nano catalyst are presented in Figs. 1 to 5.

Effect of Reaction Time on Biodiesel Yield

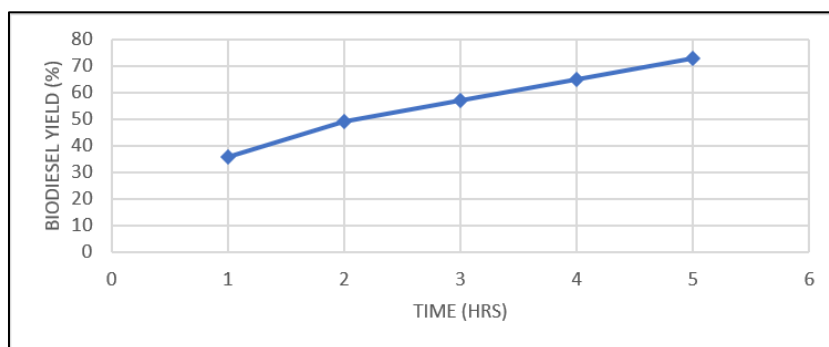


Figure 1 Effect of reaction time on biodiesel yield

Fig. 1 is a graphical presentation of the effect of reaction time on biodiesel production using the nano catalyst. Reaction time for the production of biodiesel was monitored from one (1) to five (5) hours at regular intervals. Maximum biodiesel yield of 72.98% by volume was obtained. Result shows that biodiesel yield increased progressively as time increases. During the period, other process effects remained constant.

Effect of Temperature on Biodiesel Yield

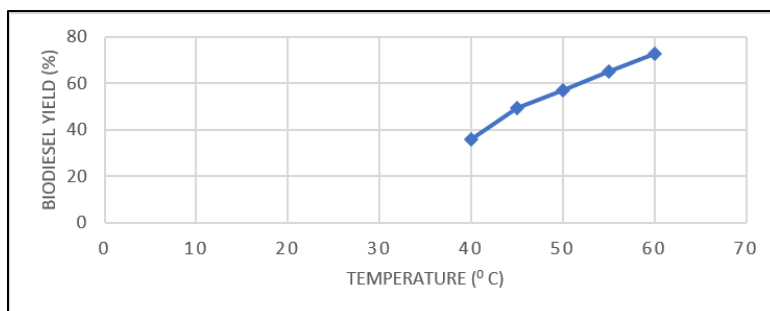


Figure 2 Effect of temperature (oC) on biodiesel yield

Fig. 2 is a representation of the effect of reaction temperature on the biodiesel yield. Temperature was varied from 40 to 60 °C at five regular intervals. Other parameters were kept constant. From the figure, biodiesel can actually be produced at temperature as low as 40°C, however, not at optimal quantity. Maximum yield by volume was 72.98%. For the catalyst, optimal temperature for biodiesel yield was 60°C. This can be attributed to the fact that methanol reacted with the sample to cause saponification, hence transesterification, thus boiling at 60°C, thereby achieving maximum activation energy at 60°C.

Effect of Methanol/Oil Molal Ratio on Biodiesel Yield

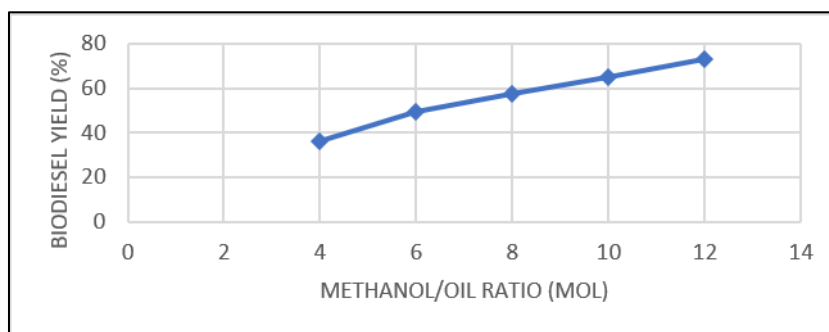


Figure 3 Effect of methanol/oil ratio (mol) on biodiesel yield

The effect of methanol and oil molal ratio on the biodiesel yield is shown in Fig. 3. Methanol to oil molal ratio was varied at five different study points (4:1, 6:1, 8:1, 10:1 and 12:1), while other study parameters were kept constant. Study time and temperature were held constant at their observed optimal conditions of 5 hrs and 60 °C respectively. Methanol and oil molal ratio, which gave the maximum yield of 72.98% was 12:1.

Effect of Catalyst Concentration on Biodiesel Yield

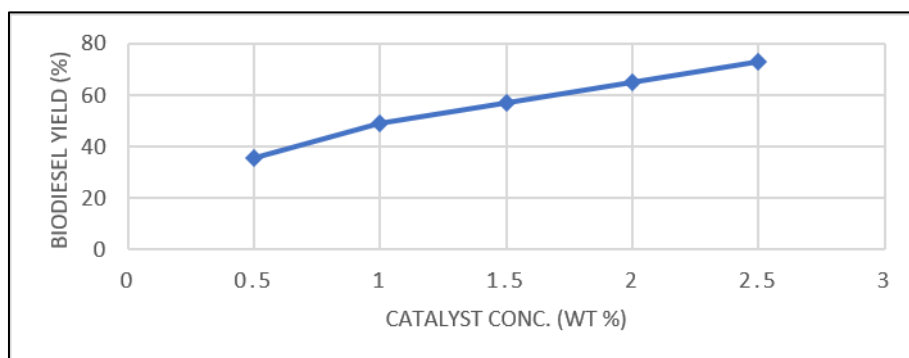


Figure 4 Effect of catalyst concentration (wt%) on biodiesel yield

The effect of catalyst, a character that enhances the rate of chemical reaction, was part of the crucial parameters studied. The nano catalyst concentration was varied at 0.5, 1, 1.5, 2, and 2.5 % weight, while the other parameters were kept constant. Conditions observed to be optimal for reaction temperature, reaction time and methanol/oil molal ratio, which have been studied were used. For the nano catalyst, concentration of 2.5% weight gave the highest yield of biodiesel of 72.98% by volume as shown in Fig. 4.

Effect of Agitation Speed on Biodiesel Yield

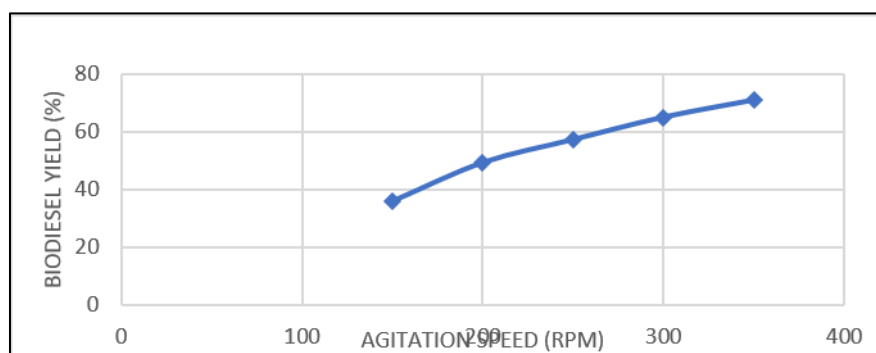


Figure 5 Effects of agitation speed on biodiesel yield

Agitation speed as an effective method ensures that non homogeneous phases are mixed. This effect mixes the reacting components (methanol and oil) and the catalyst effectively. The agitation speed was evaluated at five equal intervals of 150, 200, 250, 300 and 350 rpm, while the conditions from other parameters were kept constant. Optimal yield of 72.98% for the nano catalyst analyzed was obtained at agitation speed of 350 rpm.

4.3.2. Combined effect

The Response Surface Methodology (RSM), factorial design, was used to analyze the experimental results using the Analysis of variance (ANOVA), to determine significant interactions among the independent factors: catalyst concentration (A), methanol/oil molal ratio (B), reaction temperature (C), reaction time (D) and agitation speed (E) as the response (Y) is being produced. The result is presented in Table 3.

Table 3 ANOVA table for quadratic model using titanium oxide nano catalyst Response 1: biodiesel yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	4026.13	15	268.41	3.72	0.0065	Significant
A-catalyst conc.	51.92	1	51.92	0.7200	0.4087	
B-methanol/oil	812.25	1	812.25	11.26	0.0040	
C-temperature	524.39	1	524.39	7.27	0.0159	
D-time	16.05	1	16.05	0.2225	0.6435	
E-speed	151.99	1	151.99	2.11	0.1659	
AB	170.48	1	170.48	2.36	0.1437	
AC	183.07	1	183.07	2.54	0.1306	
AD	0.1128	1	0.1128	0.0016	0.9689	
AE	587.39	1	587.39	8.15	0.0115	
BC	866.11	1	866.11	12.01	0.0032	
BD	9.64	1	9.64	0.1336	0.7195	
BE	323.85	1	323.85	4.49	0.0501	
CD	268.66	1	268.66	3.73	0.0715	
CE	11.42	1	11.42	0.1584	0.6959	
DE	48.81	1	48.81	0.6769	0.4228	
Residual	1153.69	16	72.11			not significant
Lack of Fit	1644.37	12	137.03	0.6823	0.7271	
Pure Error	803.36	4	200.84			
Cor Total	5179.82	31				

ANOVA was applied to determine significant terms among the independent variables studied. From the Table 4, the Model F-value of 3.72 implies the model is significant. There is only 0.65% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicates that model terms are significant. In this case B, C, AE, BC are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. Though model terms highlighted include the single terms and the interaction terms, the terms of interest are the interaction terms. It depicts the interactive relationship between the terms as shown in the 3D plots. For the combined effects, methanol and temperature (BC) had the highest effect on the biodiesel yield as it took the highest F-value (12.01) while the least was catalyst concentration and time (AD), which had the lowest F-value (0.0016). By comparison, the predicted model fits the experimental data reasonably.

The results of the combined effects of process parameters on biodiesel yield are presented in Figs. 6 to 14.

Effects of catalyst concentration and methanol/oil ratio on biodiesel yield

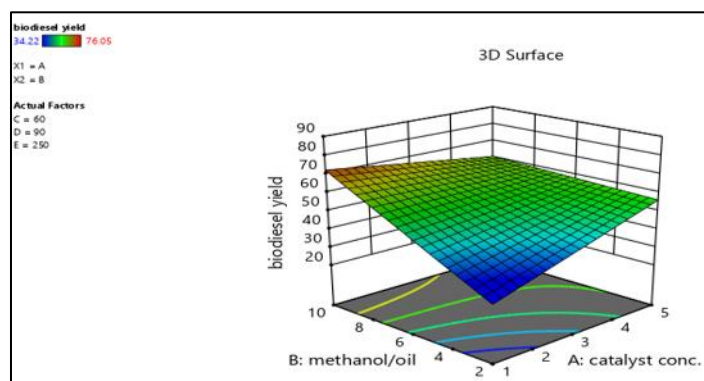


Figure 6 3D plot interaction for catalyst concentration and methanol/oil molar ratio on biodiesel yield using titanium oxide as catalyst

The RSM analysis (3D plot) automatically indicated that the predicted optimum methyl ester yield from the palm kernel shell and the cocoa pods oils blend was 76.05% at an oil-to-methanol molar ratio of 6:1, catalyst loading of 5%, time of 90 minutes, agitation speed of 250 rpm and temperature of 600C. These optimum values achieved a biodiesel yield of 76.05%. Below/above this optimum values was a decline in biodiesel yield to 34.22 and 71% respectively. From the interaction of catalyst and methanol/oil ratio presented in Fig. 6, the plot revealed there were significant interaction effects between catalyst loading and methanol/oil molar ratio of the methyl ester yield. The biodiesel increased with increase in catalyst concentration and methanol/oil molal ratio. However, the result presented in the study indicates maximum biodiesel yield at increased catalyst loading of 5% and improved methanol/oil molar ratio of 6:1.

Effect of catalyst concentration and reaction temperature on biodiesel yield

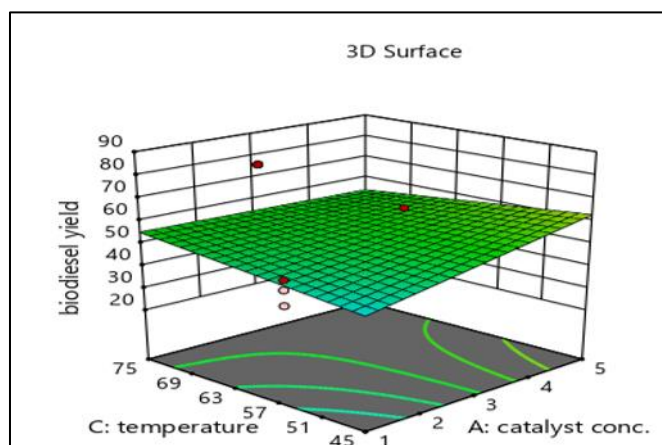


Figure 7 3D plot interaction for catalyst concentration and temperature on biodiesel yield using titanium oxide as catalyst

The 3D plot indicated that the predicted optimum methyl ester yield from titanium oxide as nano catalyst was 76.05% at a catalyst loading of 5% and temperature of 600C. From Fig. 7, the plot reveals that there are significant interaction effects between catalyst loading and temperature on the methyl ester yield. At lower ranges of catalyst concentration and reaction temperature, a relative decrease of biodiesel yield to 62% was observed. The decrease may be due to the increase in viscosity of the reaction mixture at high catalyst loading.

Effect of catalyst concentration and reaction time on biodiesel yield

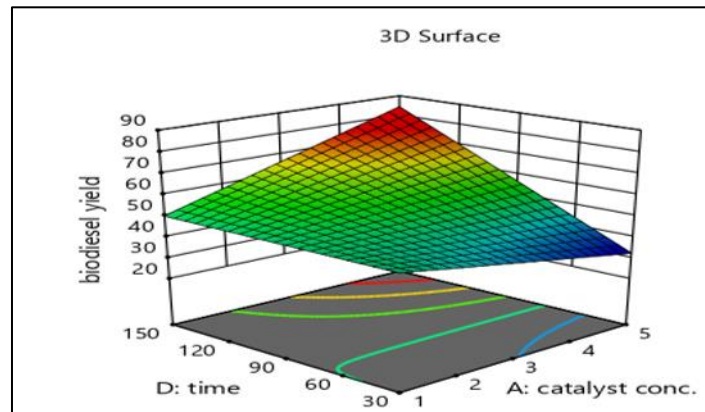


Figure 8 3D plot interaction for catalyst concentration and time on biodiesel yield using titanium oxide as catalyst

The RSM (3D) plot indicated that the predicted optimum methyl ester yield from blended palm kernel shell oil and cocoa pods oil was 76.05% as a result of the interaction between time of 90 minutes and catalyst concentration of 5%wt on the biodiesel yield. However, other parameters were kept constant. The interaction of the catalyst loading and reaction time presented in Fig. 8, revealed that there were significant interaction effects between them, on the methyl ester yield. At lower and higher ranges of catalyst concentration and reaction time, a decline in biodiesel yield of 34.22 and 49.1% was observed.

Effect of catalyst concentration and agitation speed on biodiesel yield

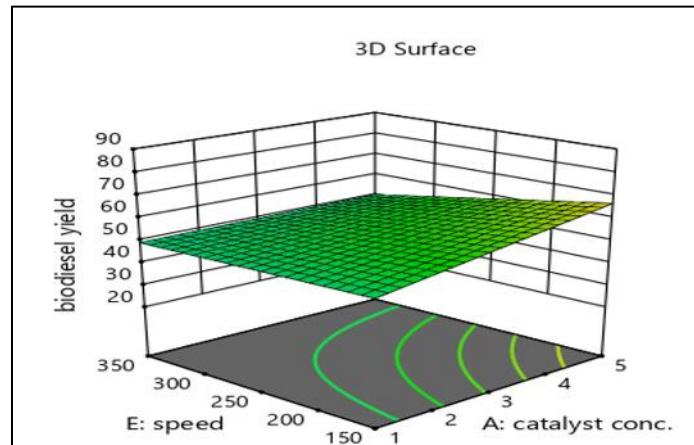


Figure 9 3D plot interaction for catalyst concentration and time on biodiesel yield using titanium oxide as catalyst

The interaction of catalyst concentration and agitation speed is presented in Fig. 9. Biodiesel increased to 65% with corresponding increase in catalyst concentration from 1 to 5 wt% and speed, from 150 to 350 rpm; but with its optimal value of 76.05% at 5 wt% and 250 rpm respectively. The increase shows that higher stirring speeds favours collision between the blends of palm kernel shell oil and cocoa pods oil mixed with alcohol molecules during the reaction.

Effect of methanol/oil molal ratio and reaction temperature on biodiesel yield

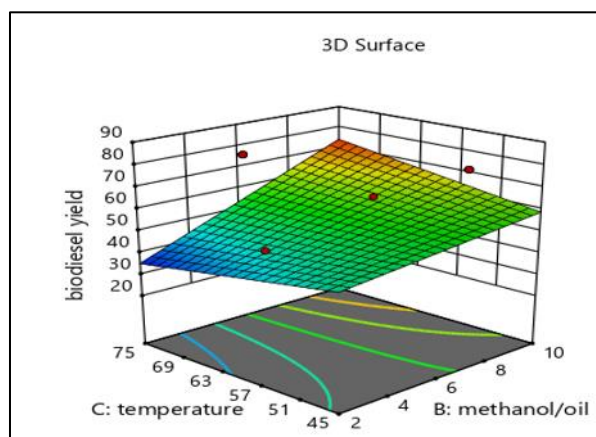


Figure 10 3D plot interaction for methanol/oil ratio and temperature on biodiesel yield using titanium oxide as catalyst

Fig. 10 shows the elliptical nature of the plot for the interactions between oil-to-methanol molar ratio and reaction temperature. It indicates that the two variables were significant to the methyl ester yield and that there was an interaction between them. Biodiesel yield increased to 76.05% with a corresponding increase in reaction temperature from 45 to 60 °C and an increase in oil-to-methanol ratio; however, excessive methanol decreased the yield. Biodiesel yield decreased to 34.22% under the conditions at a temperature and methanol/oil molar ratio of >60 °C and < 6:1 respectively. Temperature above boiling point of methanol (65°C) should be avoided because of the evaporation of methanol in the solution [40,41]; which decreases biodiesel yield.

Effect of methanol/oil molal ratio and reaction time on biodiesel yield

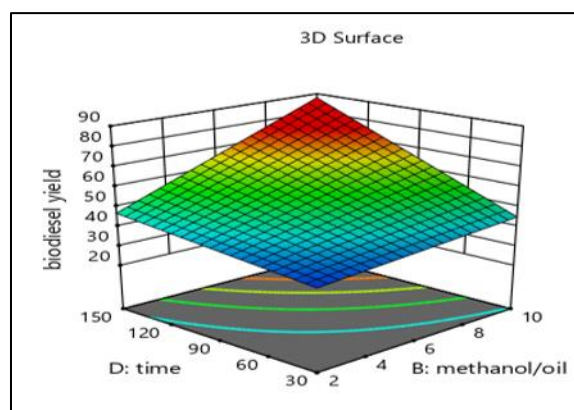


Figure 11 3D plot interaction for methanol/oil molal ratio and time on biodiesel yield using titanium oxide as catalyst

Figs. 11 shows the 3D plots for the interaction between oil-to-methanol molal ratio and reaction time. From the result, yield increased with an increase in oil-to-methanol ratio; however, excessive methanol decreased the yield, an observation that agrees with that of Velickovic et al. (2012) [48]. The predicted biodiesel yield value of 76.05% from the ANOVA was observed at reaction time of 90 minutes and methanol/oil molal ratio of 6:1 (w/w).

Effect of methanol/oil molal ratio and agitation speed on biodiesel yield

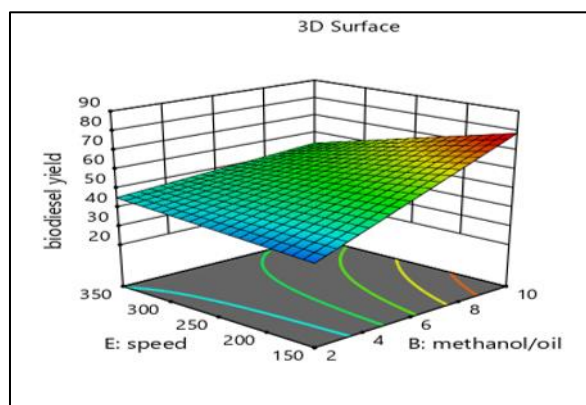


Figure 12 3D plot interaction for methanol/oil molal ratio and agitation speed on biodiesel yield using titanium oxide as catalyst

Fig. 12 shows the interaction between oil-to-methanol molar ratio and agitation speed. Maximum yield of 76.05% was obtained at methanol/oil ratio of 6:1 and agitation speed of 250rpm. The two variables were significant to methyl ester yield. Yield increased gradually with an increase in oil-to-methanol ratio. However, at about 3w/w, there was an increase in the speed to 350 rpm; which caused a decrease in the yield.

Effect of reaction temperature and reaction time on biodiesel yield

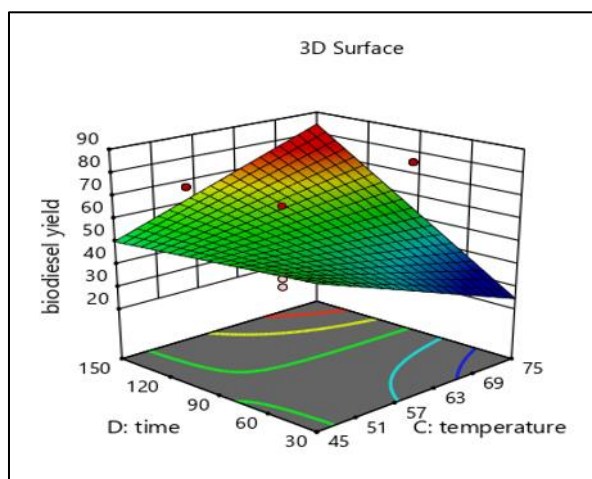


Figure 13 3D plot interaction temperature and time on biodiesel yield using titanium oxide as catalyst

Fig. 13 depicts the interaction effect between reaction temperature and reaction time on 3D plot respectively. It indicated that the biodiesel yield increased with increase in reaction time from 30 to 150 minutes and reaction temperature from 45 to 75°C respectively. At higher temperature above and below the optimal value of 60°C, a noticeable decrease in biodiesel yield to 50.1 and 30.56% was observed.

Effect of reaction time and agitation speed on biodiesel yield

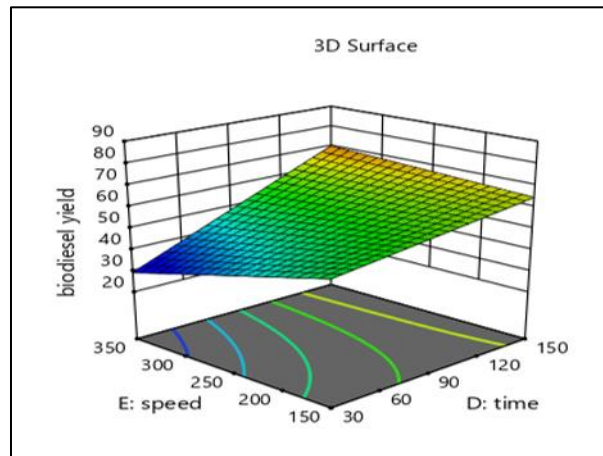


Figure 14 3D plot interaction for speed and time on biodiesel yield using titanium oxide as catalyst

Interaction effect between reaction time and agitation speed on 3D plot is shown in Fig. 14. Result indicated that the biodiesel yield of 76.05% was achieved at a reaction time of 90 minutes and an agitation speed of 250 rpm. However, at higher temperature above and below the optimal value of 90 minutes and agitation speed of 300rpm, a noticeable decrease of 64 and 28% in biodiesel yield was observed.

5. Conclusion

Biodiesel was successfully produced from blends of palm kernel shell and cocoa pods oils through transesterification process using titanium oxide as catalyst. Before the production of biodiesel, the extracted oils were blended and characterized; together with the catalyst. Results of 1.16% moisture content, 56.45 mm²s⁻¹ @40°C kinematic viscosity, 140.5 kJ/kg energy value, 194 mgKOH/kg saponification, 5.32% ash, 42.46% C, 66.56% O, 701.45 g/mol molecular weight etc. were obtained for the oil blend. For the catalyst, 2.07% moisture content, 3.97 pH, 9.84% volatile matter, 4.54% ash etc. were obtained. Five process parameters (catalyst concentration, reaction temperature, reaction time, methanol/oil molal ratio and agitation speed) were considered and varied within different ranges to anticipate biodiesel yield. An optimal design in RSM full fractional factorial was employed to achieve the optimum process parameters for the production of biodiesel using titanium oxide as catalyst. The process parameters were independent variables while the biodiesel yield was the response, known as the dependent variable. Single (experimental) and combined (predicted) effects of the process parameters on the yield were studied. A matrix design was adopted to study the combined effects. ANOVA statistics was used to predict the biodiesel yield and to understand the combined/interactive effects of the process parameters. The results are presented in 3D plots. From the ANOVA statistics, among the five (5) variables studied using titanium oxide as catalyst, the methanol/oil ratio (B) had the largest effect on the biodiesel yield as it took the highest F-value (11.26) while the least was the reaction time (D) with the lowest F-value of 0.2225 both for single effect. For the interactive (combined) effects, methanol and temperature (BC) had the highest effect on the biodiesel yield with the highest F-value of 12.01, while the least was catalyst concentration and time (AD), which had the lowest F-value (0.0016). From experimental result, the single effects of the process parameters on biodiesel gave the maximum yield of 72.98%, while the combined effects (predicted) from ANOVA gave the maximum yield of 76.05% respectively. This indicates that titanium oxide used proved to be an efficient nano catalyst for the production of biodiesel from blends of the non-edible oils from waste agro produce (palm kernel shell and cocoa pods). All results obtained were in line with the National Agency of Petroleum, Natural Gas and Biodiesel (ANP) specified limit, EN Standard, and ASTM Standard specifications for bio-oil, catalyst and biodiesel.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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